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AUSTRALIAN DEFENCE SCIENTIFIC SERVICE
MATERIALS RESEARCH LABORATORIES
MARIBYRNONG VICTORIA**

REPORT

MRL-R-674

**ENVIRONMENTAL EFFECTS ON THE MECHANICAL PROPERTIES
OF HIGH PERFORMANCE FIBRES**

J. R. Brown and N. McM. Browne

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(11) Aug '76

(12) 25p.

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 NOV 30 1976
 REQUESTED
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POSTAL ADDRESS: Chief Superintendent, Materials Research Laboratories,
 P.O. Box 50, Ascot Vale, Victoria 3032, Australia

UNCLASSIFIED

409014 VR

Printed by Defence Printing Establishment, Brunswick, Victoria.

1. Security Grading/Release Limitation: 2. Document Type/Number:

(a) Document content :
Unclassified

MRL-R-674

(b) This Page :
Unclassified

3. Document Date :

AUGUST, 1976

4. Title and Sub-Title :

ENVIRONMENTAL EFFECTS ON THE MECHANICAL PROPERTIES
OF HIGH PERFORMANCE FIBRES

5. Personal Authors : J.R. Brown and N.McM. Browne

6. Corporate Author : Materials Research Laboratories,
Maribyrnong, Victoria

7. Abstract (See reverse)

8. Descriptors :

Fibres
Properties
Ageing
Weathering
Radiation
Oxidation
Stability
Degradation

9. Cosati Classifications:

1105

11. Task Reference No.:

(DSTP-75/76)
NAV 74/08

10. Library distribution :

MRL (Maribyrnong)
MRL (S.A. Branch)
MRL (N.S.W. Branch)
WRE
Campbell Park Library

12. Sponsoring Agency
Reference :

N 433/1/5

13. Cost Code :

374700

14. Imprint :

MARIBYRNONG - MRL - 1976

A A

A B S T R A C T

Some environmental effects on the mechanical properties of the high performance fibres PBI, Nomex and Kevlar 49 are discussed. Thermal stabilities are given in terms of the changes in mechanical properties such as tensile strength, elongation at break and energy to break after thermal ageing at various elevated temperatures in inert and oxidising conditions. The effects of exposure to sunlight and ionising radiation are shown by similar measurements. The dimensional stability of the fibres at elevated temperatures and the effects of thermal ageing, exposure to sunlight and ionising radiation on the flammability properties of PBI and Nomex are discussed.

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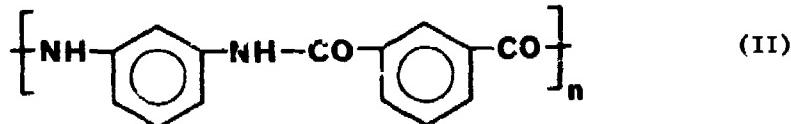
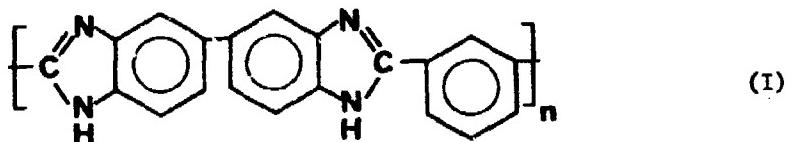
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ENVIRONMENTAL EFFECTS ON THE MECHANICAL PROPERTIES
OF HIGH PERFORMANCE FIBRES

1. INTRODUCTION

The current literature reflects the remarkable progress in the development of organic fibres that possess superior thermal and physical properties¹⁻⁶. These properties have been achieved by the incorporation of thermally stable ring structures in polymer chains and exploitation of the stabilising effects of inflexible chains, crystallinity and cross-linking.

High performance fibres were developed for specialised requirements that could not be met by existing materials. Temperature resistant fibres with suitable energy absorption characteristics that could tolerate short-term exposure to high temperatures without appreciable loss in mechanical properties were required by the United States Air Force for applications such as deceleration systems^{7,8}. In addition to retention of mechanical properties at temperatures in excess of 300°C, other outstanding textile properties have been found that have rapidly expanded the scope for possible applications. For example, the flame resistance of several temperature resistant fibres was soon appreciated and at least two, poly(2,2'-(m-phenylene)-5,5'-bibenzimidazole) or PBI (I) and poly(1,3-phenylene isophthalamide) or Nomex (II),

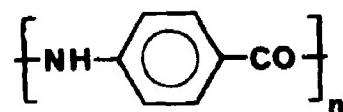


initially developed for their temperature resistant properties, owe their further development to their considerable flame resistance. Their temperature and flame resistance make them suitable for use in protective clothing. PBI has superior comfort characteristics due to its higher moisture regain of about 12% by weight compared to about 6% for Nomex.

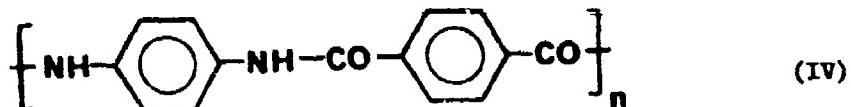
Recently, high performance fibres with more than twice the specific modulus of either glass or steel fibres and double the strength of high tenacity nylon have been developed^{4,9,10}. These high modulus fibres can be produced with a wide range of properties depending on the spinning and drawing conditions. The modulus and elongation to break can be varied to optimise their use in applications such as rigid reinforced plastics, tyre cords, and impact absorbing devices such as aircraft decelerators, safety harnesses and ballistic protection.

Unlike glass and steel, organic fibres are appreciably affected by environmental factors such as elevated temperatures, ultraviolet radiation, ionising radiation, and chemical reactants such as water, oxygen and ozone. Environmental effects include changes in composition, structure and molecular weight that can lead to degeneration of useful mechanical properties. Often two or more environmental factors combine to cause degradation; for example, oxygen and sunlight or photo-oxidation. The potential of high performance organic fibres for specialised service applications may also be governed by their stability towards one or more of these factors.

Though there are many potential high performance fibres¹⁻⁶, there are only a few currently produced commercially or even available in pilot-plant development quantities because of large-scale processing difficulties, and cost and availability of starting materials. Three such high performance fibres are PBI (I), developed by the Celanese Corporation and the United States Air Force, Nomex (II), an aromatic polyamide developed by Du Pont, and Kevlar, another aromatic polyamide recently developed by Du Pont as a high modulus, high strength fibre. It is presently available as Kevlar 49 and Kevlar 29 continuous filament fibre or fabric¹¹. Kevlar 29 has about half the modulus and double the elongation at break of Kevlar 49 and is more suitable for use in areas where high impact resistance is of primary importance, such as aircraft deceleration, safety harnesses and ballistic protection. Kevlar 49 is more suitable for use in composites such as rigid reinforced plastics. Kevlar 49 was formerly known as PRD-49 (the final production version was called PDR-49, Type III), and Kevlar 29 was formerly referred to as Fiber B and PRD-49, Type IV. It has been suggested that PRD-49, as initially introduced was poly(p-benzamide) (III), and the higher tenacity material available since 1972 is poly(1,4-phenylene terephthalamide) (IV)¹². However, pyrolysis-gas chromatography-mass spectrometry experiments conducted at these laboratories suggest that both structural units (III) and (IV) are present in Kevlar 49¹³.



(III)



This report discusses some environmental effects on the mechanical properties of PBI, Nomex and Kevlar 49 fibres. Thermal stabilities are assessed in terms of the changes in mechanical properties such as tensile strength, elongation at break and energy to break after thermal ageing at various temperatures in inert and oxidising conditions. Similar measurements illustrate the susceptibility of these fibres to degradation on exposure to sunlight and their high resistance to degradation by gamma irradiation. The dimensional stability of the fibres at elevated temperatures and the effects of thermal ageing, exposure to sunlight and ionising radiation on the flammability properties of PBI and Nomex are discussed.

2. EXPERIMENTAL

2.1 Materials

Materials evaluated were fabrics made from either continuous filament or staple fibre yarns. Nomex and Kevlar 49 (PRD-49, Type III) were supplied by the Du Pont Company. PBI was provided by the Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio. Details of each material are given in Table 1.

2.2 Environmental Exposure

Fabric samples 150 mm (warp) by 20 mm (weft) were thermally aged in a circulating air oven, either sealed in evacuated glass tubes or freely suspended. Similar samples were (a) sealed in glass tubes under vacuum and in air and irradiated with cobalt-60 gamma radiation at 30°C at a dose rate of one Mrad/hour, and (b) exposed to the weather in summer at these laboratories. After exposure, the fabrics were frayed to remove the warp yarns which were then mounted to prevent disturbance to the yarn twist.

2.3 Mechanical Properties

The yarn samples were conditioned for several days and tested at $20 \pm 2^\circ\text{C}$ and $65 \pm 2\%$ relative humidity. The yarns were strained to break at 120 mm/minute with an initial grip separation of 100 mm.

The data extracted from the recorder traces were (a) breaking force, N, (b) elongation at break, %, (c) energy to break, mJ, for a specimen length of 100 mm. These were calculated as averages from the traces of ten test specimens.

2.4 Thermal Properties

Thermomechanical analyses were conducted using a Du Pont Model 941 Thermomechanical Analyser. Limiting oxygen indices were determined by a General Electric Flammability Tester according to ASTM D 2863-74.

3. RESULTS AND DISCUSSION

3.1 Thermal Ageing

The requirements of high temperature resistance are of two types depending on intended end use. The first is retention of mechanical properties at elevated temperatures and the second is retention of mechanical properties after thermal ageing (usually in air) for extended periods of time. These requirements are related to the viscoelastic properties of the fibres and their resistance to oxidation or other degradation reactions, respectively.

While thermogravimetry shows that major chemical degradation of PBI, Nomex and Kevlar fibres occurs at temperatures greater than 400°C ¹⁴, appreciable loss in mechanical properties is evident at lower temperatures as a result of more subtle changes such as oxidation, chain fracture, crosslinking and disorientation. For example, Nomex and Kevlar retain 65-70% of their room temperature strength at 250°C after a one minute exposure at this temperature^{10,15}. Under these conditions, PBI retains about 80% of its initial room temperature strength¹⁰. The modulus and elongation at break retentions of each fibre are also 70-80%.

Resistance to thermal ageing or changes in room temperature properties after exposure to elevated temperatures for extended periods is, in many cases, a more important measure of the thermal stability. Changes in breaking force, elongation at break and energy to break of Nomex, Kevlar 49 and PBI yarns have been determined as a function of exposure period at various temperatures in vacuum and air.

Figure 1 shows these property retentions for undyed Nomex. Extended exposure (300 hours) at temperatures up to 200°C in vacuum and air has no significant effect on the mechanical properties. Nomex has been reported to retain about 80% of its strength after exposure to air at 177°C for several thousand hours⁸. After exposure at 250°C for 300 hours in vacuum and air, Nomex retains about 80% of its initial room temperature strength and elongation at break. The energy to break retention under these conditions is about 60%. There are no discernible effects of air on the changes in mechanical properties on ageing at 250°C .

After ageing for 300 hours at 300°C in air and vacuum, the mechanical properties of Nomex are markedly reduced. The strength and elongation at break retentions are similar (~35%) and the energy to break is reduced to about 8% of its initial value. The loss in mechanical properties after shorter exposures at 300°C is greater in vacuum than it is in air. This is probably due to hydrolytic degradation of the amide bond by water which is strongly absorbed and not completely removed in the evacuation process prior to sealing of the samples. During exposure at temperatures in excess

of 300°C, the mechanical properties are rapidly reduced. This is attributed to the glass transition in the vicinity of 300°C¹⁴ in addition to thermal degradation of the fibres.

Figure 2 shows the changes in mechanical properties of Kevlar 49 on thermal ageing at 100°C - 300°C in air and vacuum. Extended exposure at 100°C does not alter the mechanical properties while at 150°C, the strength, elongation at break and energy to break are reduced to 70-80% of their original values.

After exposure at 200°C for 300 hours in air and vacuum, the mechanical properties of Kevlar 49 are appreciably reduced; strength, elongation at break and energy to break retentions are about 40%, 50-60% and 20%, respectively. There is no marked effect of air on the degradation at 200°C. A faster degradation after shorter exposures in vacuum is attributed to amide hydrolysis.

The mechanical properties are rapidly reduced on ageing at 250°C in air and vacuum. Strength, elongation at break and energy to break retentions after 300 hours exposure are about 25%, 45% and 12%, respectively. Almost all the loss in mechanical properties occurs within the first 50 hours exposure.

Above 250°C, additional effects of oxidation are appreciable. Exposure at 300°C in vacuum rapidly reduces the strength, elongation at break and energy to break to about 20%, 30% and 5%, respectively of their original values, while exposure at 300°C in air rapidly reduces the mechanical properties to zero. As for Nomex, exposure of Kevlar 49 in both air and vacuum at temperatures in excess of 300°C rapidly destroys the mechanical properties. This is again attributed to the glass transition in the region of 300°C¹⁴ and concurrent oxidation and thermal degradation.

These results show that Kevlar 49 is more susceptible to thermal ageing than Nomex. Kevlar 49 owes much of its high strength to its high degree of molecular order and high intermolecular forces (hydrogen bonding)¹⁶, and a rapid reduction in mechanical properties on thermal ageing above 200°C may be attributed to partial disruption of secondary bonding, although there was no significant change in the modulus. It has been reported that the long term use temperature in air should not exceed 160°C¹⁷. The apparently higher susceptibility of Kevlar 49 to oxidation at elevated temperatures has also been established by thermomechanical analysis (TMA) of Kevlar 49 and Nomex¹⁴. This may be attributed to a low initial molecular weight of Kevlar 49 (degree of polymerisation about 100 which corresponds to an average of 200 amide links per molecule¹⁰) compared with Nomex ($M \sim 39,000$, corresponding to an average of 320 amide links per molecule¹⁸). A similar yield of chain fractures due to thermal oxidation may in this case have a greater effect on the mechanical properties of Kevlar 49.

Figure 3 shows the effects of thermal ageing in vacuum and air on the mechanical properties of PBI continuous filament yarn. Figure 4 gives similar data for PBI undyed staple fibre yarn. The most significant factor of both yarns is the remarkable stability to thermal ageing in vacuum. After 300 hours exposure in vacuum at temperatures up to 350°C, there are no significant changes in the mechanical properties of the fibres.

Heat ageing in air for extended periods at temperatures up to 200°C has little effect on the mechanical properties. After exposure at 250°C in air for 300 hours, the mechanical properties of PBI continuous filament yarn show appreciable reductions; retentions of strength, elongation at break and energy to break are about 80%, 70% and 60%, respectively. After similar exposure, PBI undyed staple fibre yarn shows smaller reductions; retentions of strength, elongation at break and energy to break are about 90%, 97% and 90%, respectively. In the range 250°C - 300°C in air, the mechanical properties of each fibre are markedly reduced. After 300 hours at 300°C in air, the strength and energy to break of each fibre are reduced to less than 5% of their original values and the elongation at break retention is about 15%.

It has been reported that the characteristic benzimidazole infrared absorption bands in PBI become indistinguishable and a nitrile absorption band at 2236 cm⁻¹ appears after exposure at 300°C for seven hours in air¹⁹. These observations and the marked loss in mechanical properties after exposure in air at this temperature indicate that the benzimidazole unit is very susceptible to oxidation.

Figure 5 shows the changes in mechanical properties on thermal ageing of dyed, stabilised PBI staple fibre yarn in air and vacuum. (The stabilisation process is discussed in section 3.2). There are no significant changes in mechanical properties after 300 hours exposure in vacuum at temperatures up to 300°C. At 350°C, the strength, elongation at break and energy to break retentions are about 85%, 75% and 50%, respectively after 300 hours exposure. In air the stabilised material shows a greater susceptibility to thermal oxidation than the untreated material. After 300 hours exposure at 200°C, 250°C and 300°C in air, the strength retentions are about 80%, 50% and zero, respectively. Corresponding elongation at break retentions are 76%, 33% and zero, respectively, and energy to break retentions are 50%, 10% and zero, respectively.

3.2 Flammability and Thermal Shrinkage

The relatively high thermal stability of Nomex and PBI fibres and their fabricated forms has led to their use as protective clothing in hazardous environments, where short-term protection against heat and flames is of primary concern. Nomex is now used extensively and PBI on a smaller scale for this purpose. Such apparel must provide an effective thermal barrier, be non-flammable in air and non-melting, have good dimensional stability at high temperatures and be durable and comfortable. Nomex and PBI have limiting oxygen indices (LOI) of about 28 and 40, respectively, and do not melt but carbonise extensively on exposure to heat as shown by thermogravimetry weight retentions¹⁴. Thermal ageing of Nomex and PBI for 300 hours at 300°C has no significant effect on the LOI values of each fabric.

Appreciable shrinkage of Nomex and PBI occurs above the glass transition temperatures due to increased chain mobility and a relaxation of the highly strained anisotropic configuration of the drawn fibres. This is of course undesirable, as shrinkage results in closer contact between a protective garment and the wearer. Consequently much research has been conducted on improving the dimensional stability of Nomex and PBI. The

flammability and dimensional stability of Nomex have been reported to be improved by halogenation in an inert atmosphere at 300°C and by treatment with organophosphorus compounds, without any significant loss in mechanical properties²⁰. Similarly the flammability of PBI has been improved (LOI ~ 53), as well as the dimensional stability, by treatment with 5% chlorosulphonic acid in phosphorus oxychloride solution followed by heating to 340°C and neutralisation²¹. This is the stabilised material whose mechanical properties on thermal ageing have been discussed in this report. It is believed that the treatment provides - SO₂ - crosslinks between polymer chains and retards relaxation of the fibres above the glass transition temperature. The treatment decreases the stability of PBI to thermal ageing as illustrated by a comparison of the retention of mechanical properties of stabilised and untreated material. However the dimensional stability of stabilised PBI at high temperatures is greatly improved. Figure 6 shows the shrinkage of Nomex, PBI staple fibre yarn and stabilised PBI staple fibre yarn as well as Kevlar 49 measured by thermomechanical analysis. Figure 7 illustrates the shrinkage of untreated and stabilised PBI fabric after heat ageing in air for 100 hours at 400°C.

3.3 Outdoor Exposure

In contrast to their relatively high thermal stability, Kevlar 49, Nomex and PBI fibres are susceptible to rapid degradation on outdoor exposure. Figure 8 shows the results of mechanical tests on Kevlar 49, Nomex and PBI fibres exposed outdoors at these laboratories. In each case, there is a rapid fall in strength, elongation at break and energy to break that compares unfavourably even with the behaviour of unstabilised nylon 6,6. After four months outdoor exposure, the mechanical properties of each fibre have been reduced to less than 20% of their original values.

PBI has been shown to be a photochemically stable structure²²; its high rate of photodegradation is related to the intense light absorption in the solar spectral region. Outdoor exposure in controlled atmospheres resulted in a marked decrease in mechanical properties in the presence of air, while in vacuum or saturated water vapour atmospheres, the mechanical properties showed no appreciable change²². From these results, the degradation is one of photo-oxidation. PBI also undergoes a rapid darkening in colour on outdoor exposure as a result of the combined action of oxygen and ultraviolet radiation. The light-fastness of the dyed material is poor and it has been shown that stabilisation of the ground colour on exposure to light is the most important factor in attaining light-fastness²³.

Nomex and Kevlar 49 are similar to PBI in that they also show intense absorptions in the near ultraviolet (up to 370 nm), and can also be regarded as photochemically stable structures considering the large amount of energy absorbed. The degradation of Nomex on exposure to sunlight has been shown to be due to photo-oxidation¹⁸; the mechanism of photodegradation in air and vacuum has been studied by Wiles et al^{24,25}.

The intense absorption of these high performance fibres at wavelengths greater than 350 nm has suggested that for efficient photoprotection, suitable ultraviolet absorbers must be concentrated at the fibre surface. Any approach of attaching absorbers to the surface, in order to be successful, must overcome the susceptibility to photo-oxidation and also not cause any appreciable changes in fundamental properties such as high strength, flame resistance and thermal stability.

Flammability properties of each material were not appreciably affected after four months outdoor exposure. The LOI values for Nomex, Kevlar 49 and undyed PBI, after conditioning at $20 \pm 2^\circ\text{C}$ and $65 \pm 2\%$ relative humidity, showed no significant changes on outdoor exposure. A small decrease in LOI, 53 to 50, occurred for dyed, stabilised PBI. Day and Wiles²⁶ observed a small decrease in LOI, 1.4 - 2%, in Nomex which had been irradiated in a xenon arc Weather-Ometer. It can be concluded that the loss in flame resistance on outdoor exposure is insignificant compared with the marked loss in mechanical properties.

3.4 Exposure to Ionising Radiation

The incorporation of aromatic groups into the structure of polymers greatly reduces their sensitivity to ionising radiation. The G values for crosslinking and main-chain scission are considerably less than for polymers which do not contain aromatic structures. Thus aromatic polyamides and polybenzimidazoles would be expected to combine high ionising radiation resistance with their desirable thermal properties because of their aromatic content. Nomex has been reported⁷ to be resistant to gamma radiation when no appreciable changes in mechanical properties occurred after 14 Mrad, although the term radiation resistance is usually associated with property retention after much higher doses.

Table 2 gives the per cent retentions of strength, elongation at break and energy to break of Nomex, Kevlar 49 and PBI fibres after exposure to gamma irradiation in vacuum and air. The mechanical properties of PBI are not reduced significantly after 200 Mrad in air and vacuum. The elongation at break and energy to break of the PBI staple fibres have in fact increased significantly. The mechanical properties of Nomex and Kevlar 49 are virtually unaffected after 200 Mrad in vacuum. In air after 200 Mrad, Nomex shows a small reduction in mechanical properties while Kevlar 49 is affected to a larger extent.

The LOI values of each material after 100 and 200 Mrad were unchanged; thus as in the cases of thermal ageing and outdoor exposure, the flammability properties are not significantly affected by exposure to high doses of ionising radiation.

4. CONCLUSIONS

Thermal stabilities of Nomex, Kevlar 49 and PBI yarns have been assessed by changes in tensile strength, elongation at break and energy to break after thermal ageing at elevated temperatures in inert and oxidising conditions. Temperature limits for extended use in air and vacuum are about 150°C for Kevlar 49 and 200°C for Nomex. PBI has a temperature limit of about 200°C for extended use in air, but in vacuum it is considerably higher at about 350°C . Similar mechanical measurements show that the three materials are susceptible to degradation by sunlight and are resistant to degradation by gamma radiation.

Thermomechanical analysis of the fibres provides a useful measure of their relative dimensional stabilities at elevated temperatures and the

effects of thermal ageing, exposure to sunlight and ionising radiation on the flammability properties of PBI and Nomex are shown to be negligible.

5. ACKNOWLEDGEMENT

The assistance of Dr. B.C. Ennis in providing the thermomechanical analysis results is acknowledged with thanks.

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TABLE 1

MATERIAL DETAILS

Material	Yarn Designation (BS 946:1970)	Tenacity (mN/tex)	Elongation at Break (%)	Specific Energy to Break (mJ/tex)
PBI continuous filament	R 136 dtex f 1 Z 105	280	17.7	3.7
PBI undyed staple fibre	29.1 tex Z 615	215	19.6	2.9
PBI dyed stabilised staple fibre	19.6 tex Z 755	150	19.7	2.0
Nomex undyed staple fibre	R 38.5 tex S 740/2 Z 435	290	25.6	4.8
Kevlar 49 continuous filament	R 226 dtex f 1 Z 43	1540	2.7	2.0

TABLE 2

EFFECT OF GAMMA IRRADIATION IN AIR AND VACUUM
ON THE MECHANICAL PROPERTIES OF NOMEX,
KEVLAR 49 AND PBI FIBRES

Material	Dose (Mrad)	Property Retention (%)					
		In Air		In Vacuum			
Nomex, undyed staple fibre	Tensile Strength	Elongation at Break	Energy to Break	Tensile Strength	Elongation at Break	Energy to Break	
	100	85	101	80	107	107	114
Kevlar 49 continuous filament	200	90	94	80	103	104	103
	100	77	87	62	107	94	105
PBI, continuous filament	200	35	52	17	96	89	95
	100	96	105	95	100	105	108
PBI, undyed staple fibre	200	103	111	109	98	111	103
	100	102	127	117	110	130	138
PBI, dyed stabilised staple fibre	200	107	123	124	109	132	134
	100	100	133	132	103	135	128
	200	98	126	122	104	129	132

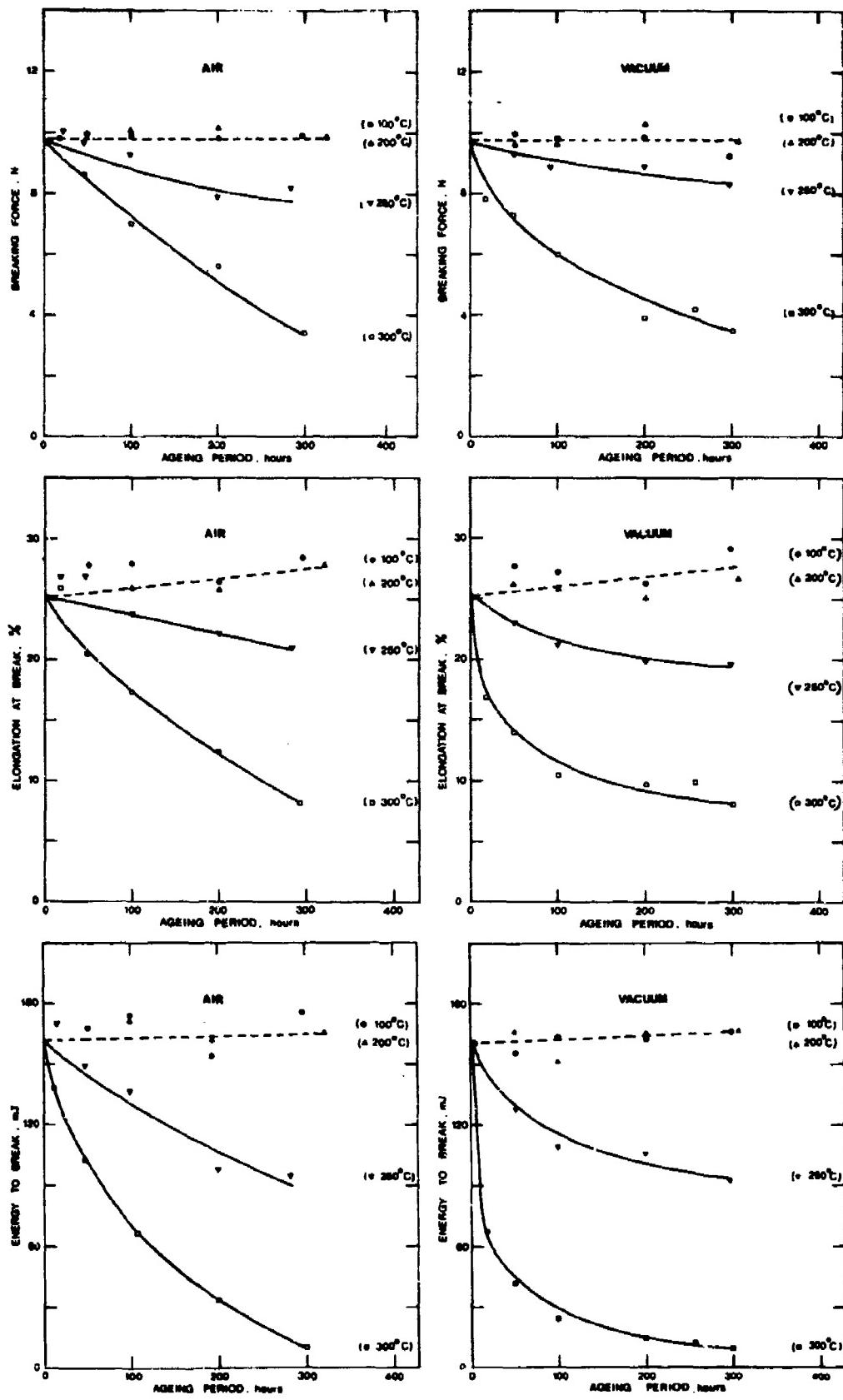


FIG. 1 - Mechanical properties of Nomex staple yarn vs exposure period at various temperatures.

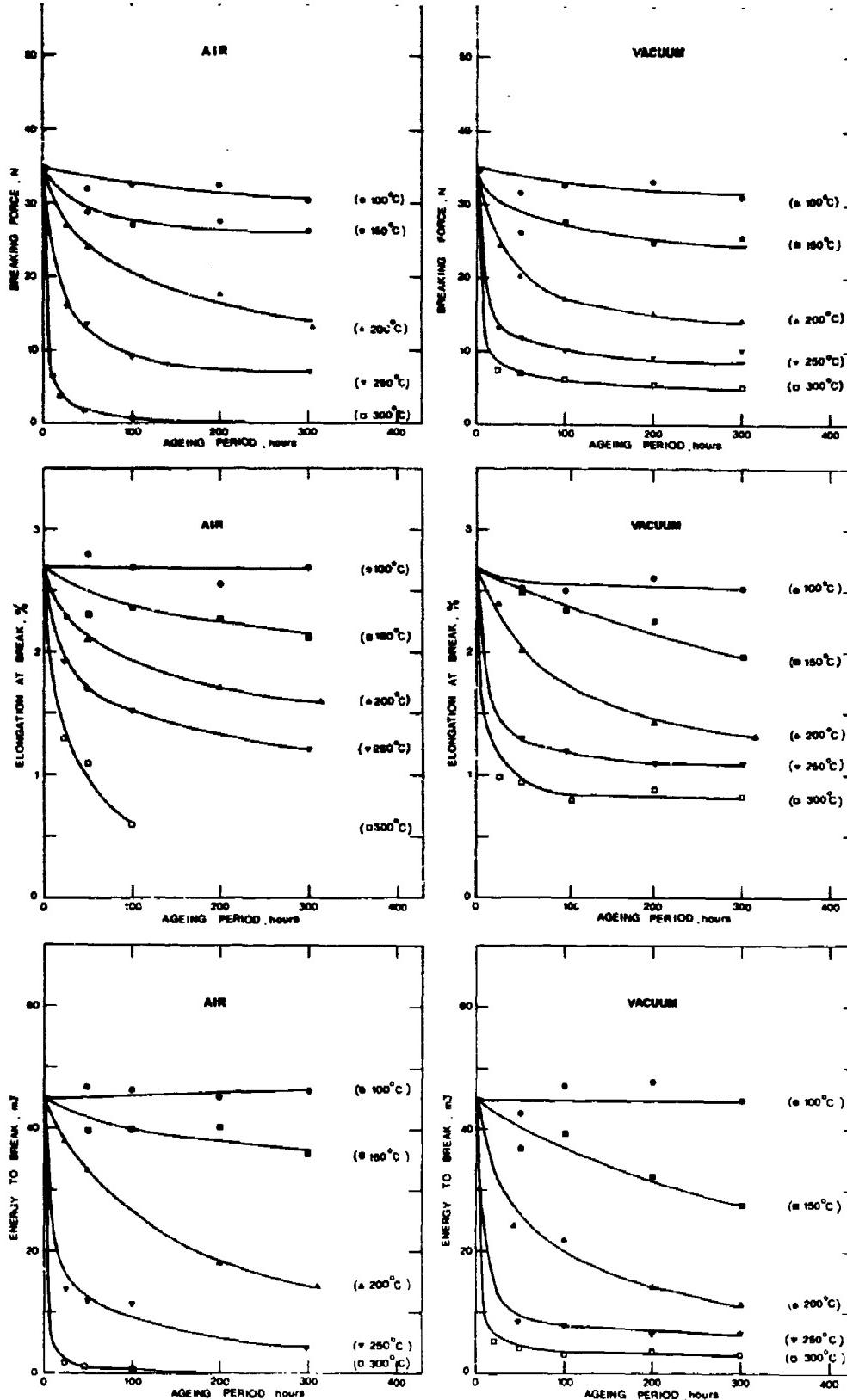


FIG. 2 - Mechanical properties of Kevlar 49 continuous filament yarn vs exposure period at various temperatures.

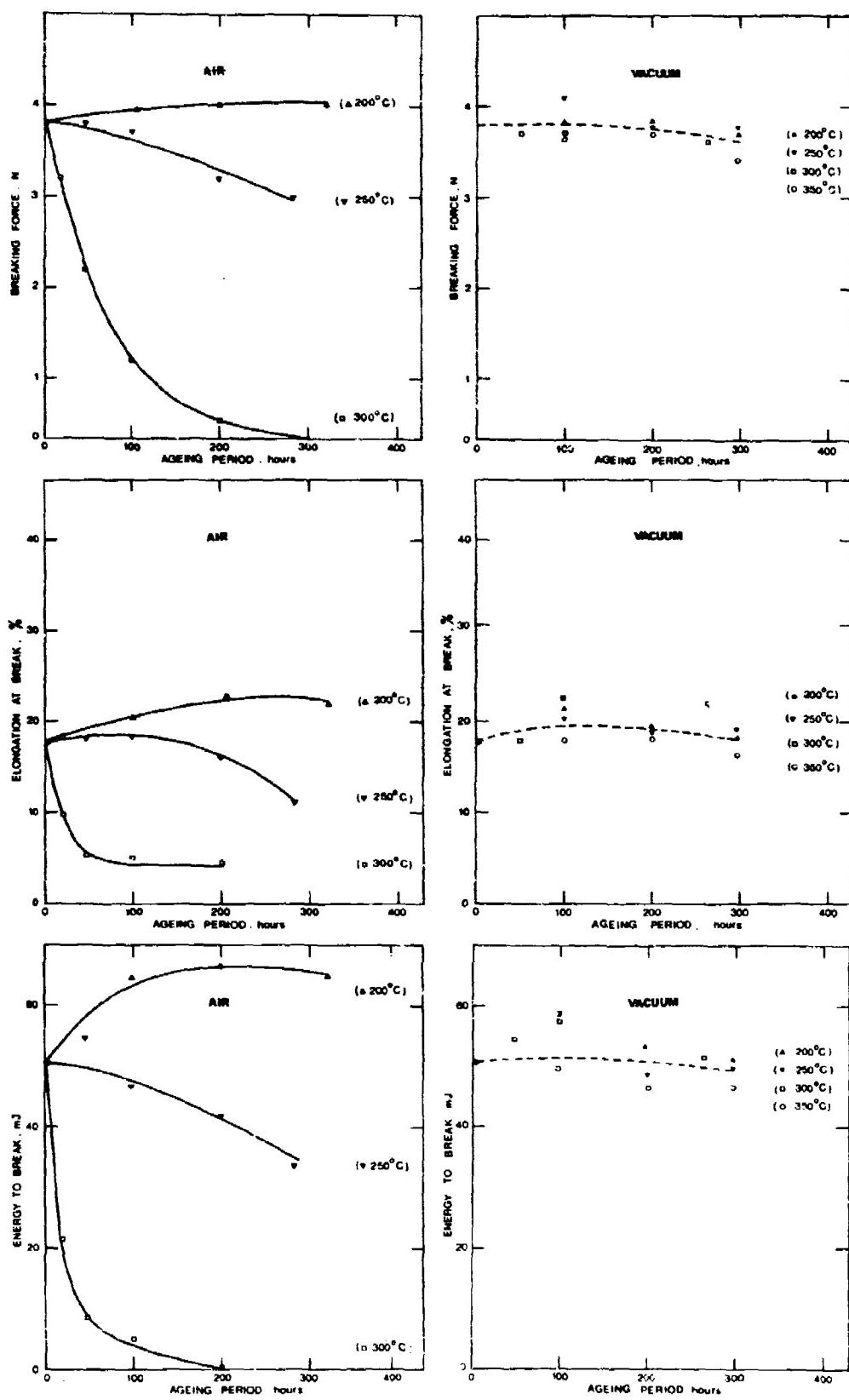


FIG. 3 - Mechanical properties of PBI continuous filament yarn vs exposure period at various temperatures.

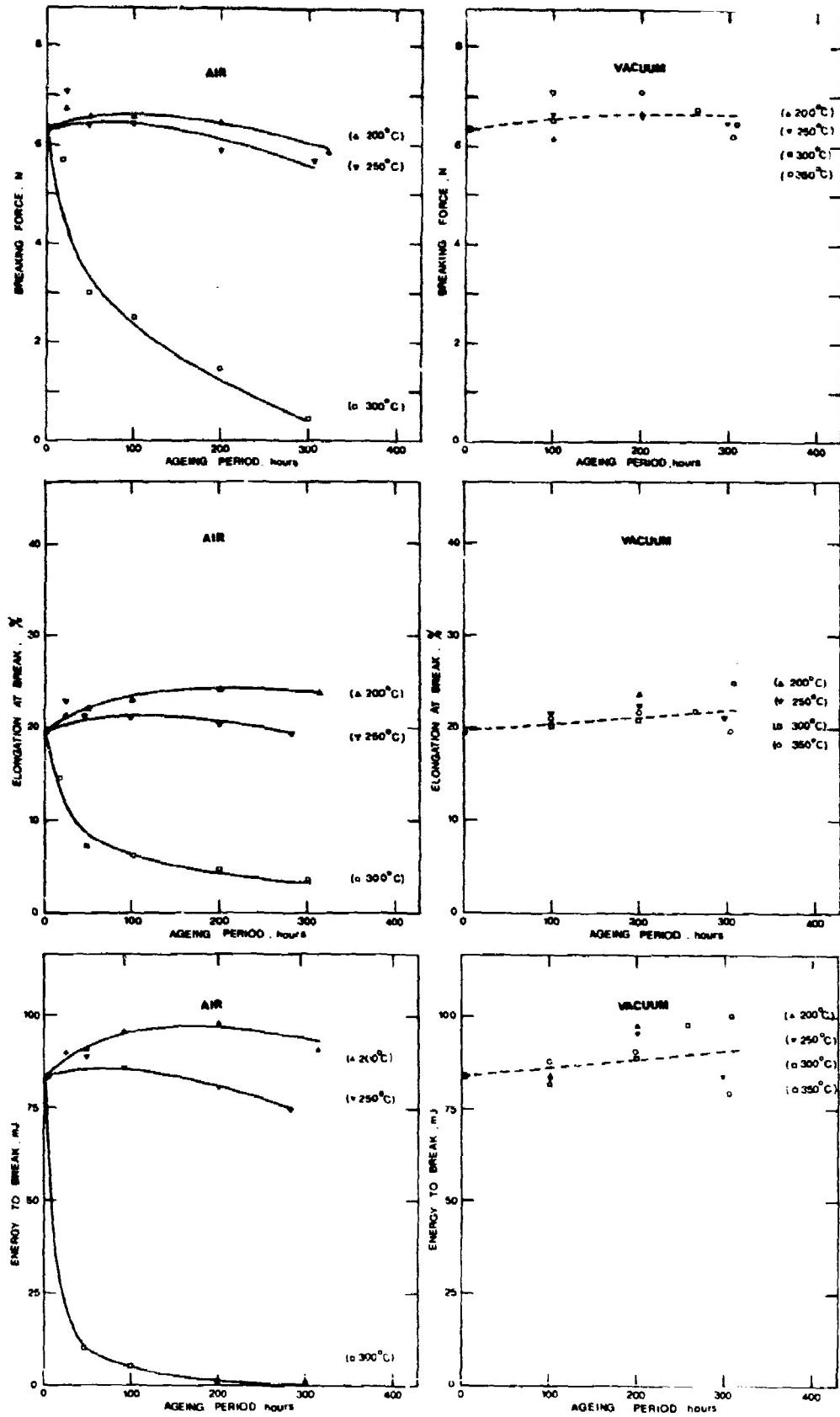


FIG. 4 - Mechanical properties of PBI staple yarn vs exposure period at various temperatures.

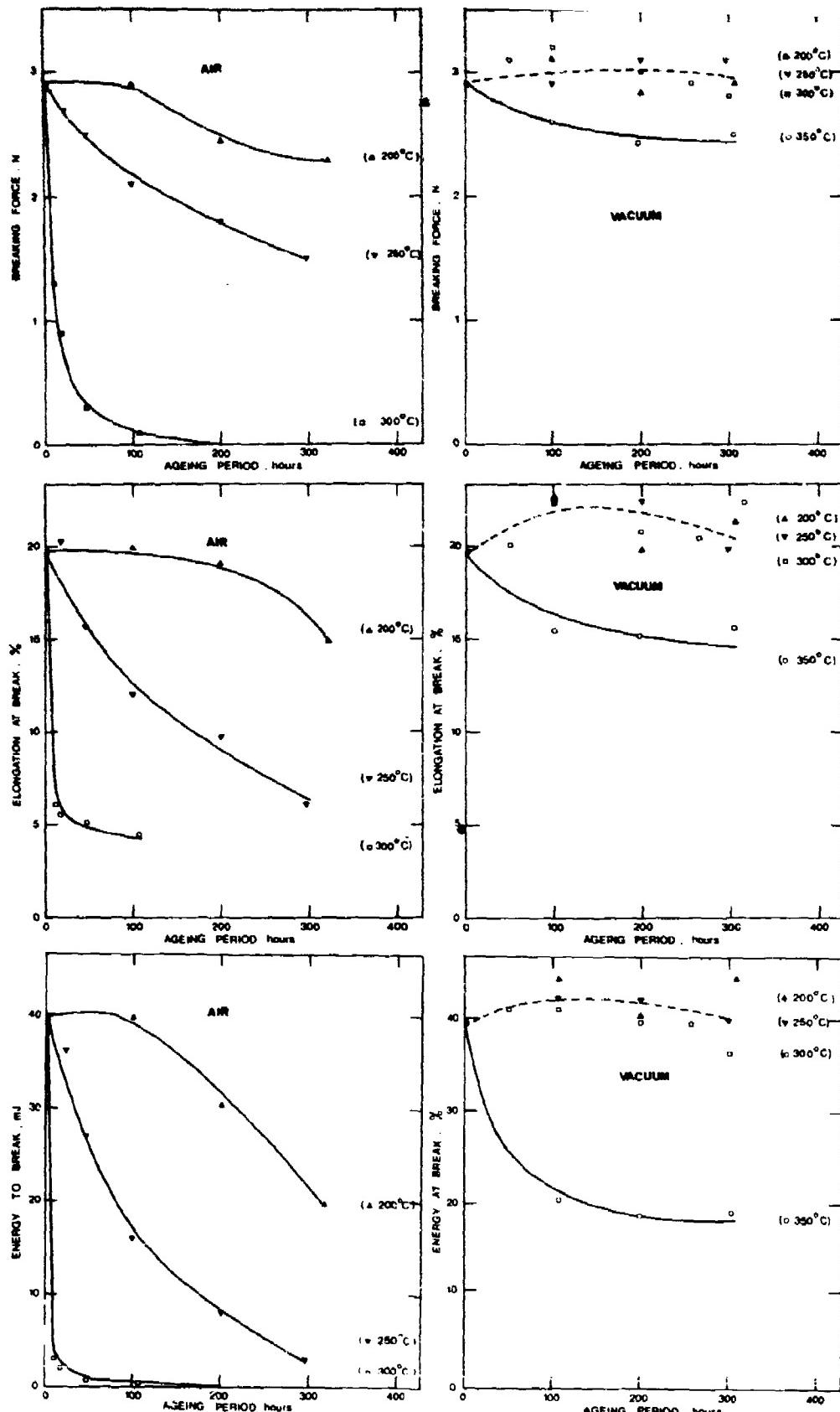


FIG. 5 - Mechanical properties of stabilised PBI staple yarn vs exposure period at various temperatures.

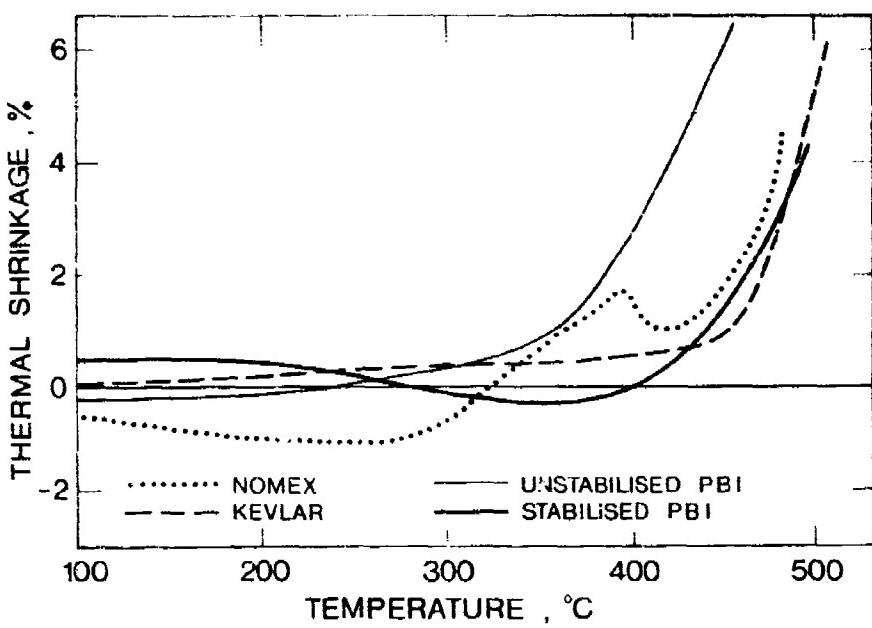


FIG. 6 - Thermal shrinkage of PBI, Nomex and Kevlar 49 by thermomechanical analysis. Du Pont TMA 941, load 0.2 g, heating rate 20°C/minute.



UNEXPOSED



STABILISED FABRIC



UN STABILISED FABRIC

FIG. 7 - Thermal shrinkage of unstabilised and stabilised PBI staple fabric after exposure at 400°C for 100 hours in air.

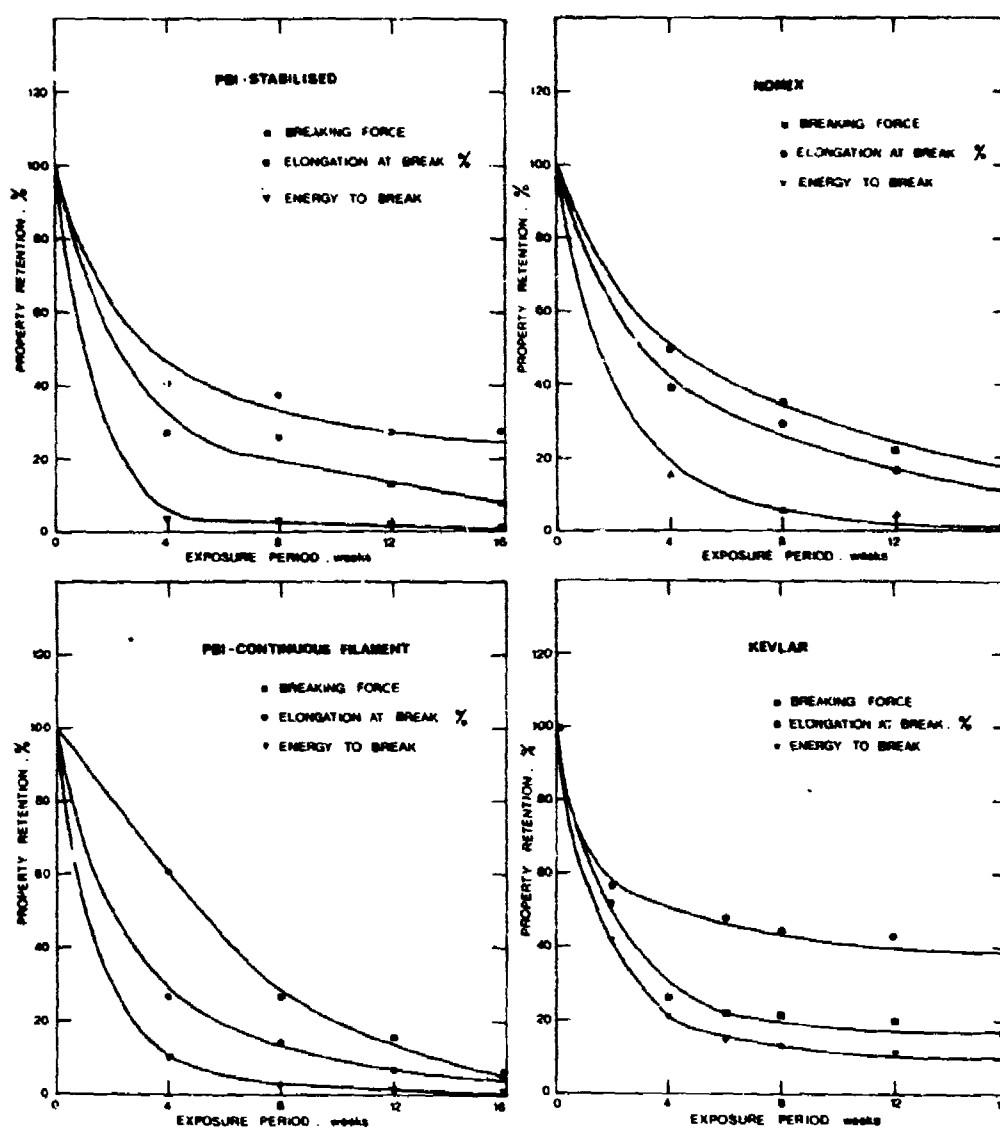


FIG. 8 - Retention of mechanical properties of Nomex, Kevlar 49 and PBI yarn on outdoor exposure.

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